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MASSACHUSETTS INSTITUTE OF TECHNOLOGY
LINCOLN LABORATORY

InP MATERIALS

ANNUAL TECHNICAL SUMMARY REPORT TO THE ROME AIR DEVELOPMENT CENTER

(10) Gerald W. Iseler

1 OCTOBER 1980 — 30 SEPTEMBER 1981

(12) 28

ISSUED 24 NOVEMBER 1981

(19) 2306

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ABSTRACT

This report covers the work on InP materials carried out with support of the Department of the Air Force during the period 1 October 1980 through 30 September 1981. A part of this support was provided by the the Rome Air Development Center.

The current objectives of ~~the~~ program are to improve the yield of high-purity polycrystalline InP as source material for crystal growth and to optimize the liquid-encapsulated Czochralski (LEC) method in order to grow crystals with low dislocation density and uniform dopant concentration.

We have synthesized 35 ingots of InP with carrier mobilities at 77 K as high as 6.8×10^4 $cm^2V^{-1}s^{-1}$. Using this charge material we have grown 26 nominally undoped LEC boules with mobilities at 77 K up to 4.2×10^4 $cm^2V^{-1}sec^{-1}$ and an additional 70 boules doped with Sn, Fe, S, Cd, Zn, or Cr.

We have grown LEC crystals that are nearly dislocation-free up to a diameter of 20 mm, wihout Zn or S doping, by increasing the thickness of the B_2O_3 encapsulant layer in order to reduce the radial temperature gradients in the growing boule. Although this procedure also reduces the vertical temperature gradient at the seed-melt interface, we ~~have been~~ were able to obtain reproducible twin-free growth in the lower gradient by modifying the pulling rod and its rotation mechanism to reduce system vibration.

By means of x-ray topography and CO_2 laser transmission studies we have found that doped LEC crystals contain prominent growth striations, probably produced by random convection currents in the melt, that are associated with abrupt changes in dopant concentration.

Measurements of infrared transmission at 10.6 μm have been made on p-type InP samples to determine the free-carrier absorption due to holes. The absorption cross section is found to be much higher for holes than for electrons.

INTRODUCTION

The goals of the InP materials program at Lincoln Laboratory are the development of reliable techniques for preparing high-quality InP single crystals of controlled electrical properties and the utilization of these techniques to produce crystals needed for research on optoelectronic devices such as GaInAsP/InP diode lasers and detectors.

Our program consists of two components: synthesis of InP from the elements and crystal growth by the liquid-encapsulated Czochralski (LEC) method. Synthesis, which is accomplished by directional solidification of In-rich solutions under controlled P pressure, is carried out in order to assure an adequate supply of polycrystalline charge material with the purity desired for LEC growth, since such material is not consistently available from commercial sources. In the area of LEC growth, by adjusting the vertical and radial temperature gradients in the vicinity of the seed-melt interface we have succeeded in reproducibly growing twin-free crystals that are nearly dislocation-free up to a diameter of ~ 20 mm, without Zn or S doping. By means of x-ray topography and CO_2 laser transmission studies we have found that doped LEC crystals contain prominent growth striations, probably produced by random convection currents in the melt, that are associated with abrupt changes in dopant concentration. We are now refining the growth conditions to increase the maximum diameter of dislocation-free material and reduce dopant inhomogeneity without increasing the occurrence of twinning.

SYNTHESIS

Synthesis of InP from the elements is carried out in a manner very similar to that reported for the previous contract period. Figure 1 shows a diagram of the fused-silica synthesis ampoule, drawn approximately to scale, together with a typical temperature profile of the resistance-heated clamshell furnace. The ampoule is fabricated with the large end open, then etched in aqua regia, vacuum baked at ~ 1000°C, and loaded with a charge of red P at the closed end and a charge of 50- or 100-g In ingots at the open end. The In ingots are placed in a fused-silica boat 21 cm long that is fabricated from 44 mm ID tubing. The P is used as received, while the In is etched in ultra-high-purity hydrochloric and nitric acids and rinsed with deionized water. The P and In charges now typically weigh 180 and 700 g, respectively. They are separated by means of an evacuated fused-silica plug that closely fits the ampoule with a clearance of less than 2 mm. The plug is used to reduce the transfer of heat from the hot zone to the P reservoir when the ampoule is heated to the synthesis temperature, in order to prevent explosions that could result from overheating the P charge. After the In and P have been loaded, a flared fused-silica cap is fused to the open end of the ampoule. The ampoule is evacuated with a vac-ion pump to a pressure of less than 10^{-6} Torr, while being heated with resistance tape to ~ 100°C, and finally sealed off.

The ampoule is tranferred to the synthesis furnace, which is then heated to give a profile like that plotted in Fig. 1. After the desired temperature is achieved, usually in about 6 hours, the ampoule is pulled through the furnace at a rate of about 2.5 cm per day. A control thermocouple tied to a silica loop at the end of the ampoule containing the P, as shown in Fig. 1, is used for maintaining the temperature at that point on the ampoule constant

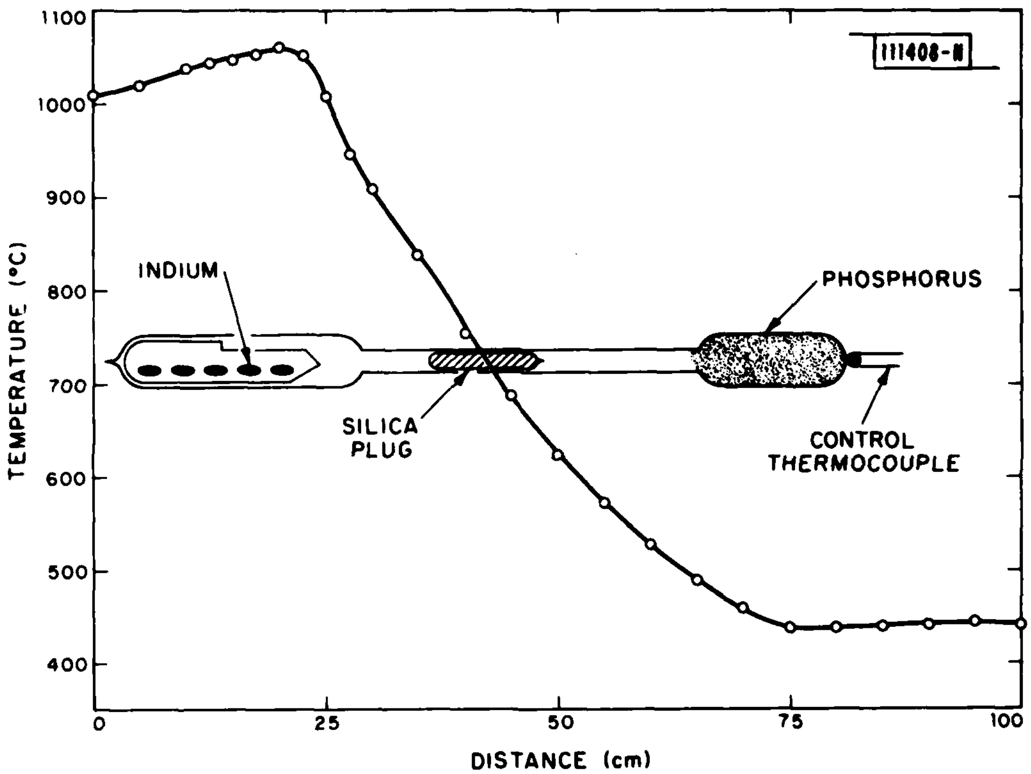

Fig. 1. Fused-silica ampoule and typical temperature profile for synthesis of InP.

during each run. This temperature has been varied between 430 and 460°C from run to run. The temperature profile shown in Fig. 1 was obtained with a loaded and sealed synthesis ampoule in the furnace just before the ampoule was pulled through the furnace. The measuring thermocouple was contained in a 6 mm ID silica tube running the length of the furnace and lying just underneath the ampoule. It should be noted that the temperature of the melt in the boat may have been quite different from the value shown on the profile because there are large temperature gradients in the vicinity of the boat; thermocouples placed at the same axial position but at various radial or vertical positions around the boat indicate temperature differences as large as 25°C. During each run the peak temperature of the furnace is maintained constant with a stationary thermocouple. This temperature has been varied between 1035 and 1075°C from run to run. After synthesis is complete, which requires about eight days, the furnace power is turned off and the ampoule is furnace cooled.

This synthesis procedure yields ingots that are highly polycrystalline, but many of them contain grains large enough to permit single-crystal rectangular bars about 10 x 2 x 1 mm^3 to be cut out for Hall coefficient and resistivity measurements. The ingots all contain In inclusions, but the concentration of elemental In is usually quite low except at the last-to-freeze end.

During FY81 (October 1980 through September 1981) we have synthesized 35 InP ingots from 6 9's In and P purchased from M.C.P. Electronics, Ltd. Electrical measurements have been made at 300 and 77 K on one sample from each of nine ingots. Each sample was taken between one and three inches from the first-to-freeze end of the ingot, where the grain size was largest. All the samples are n-type. Figure 2 shows the distribution of electron Hall

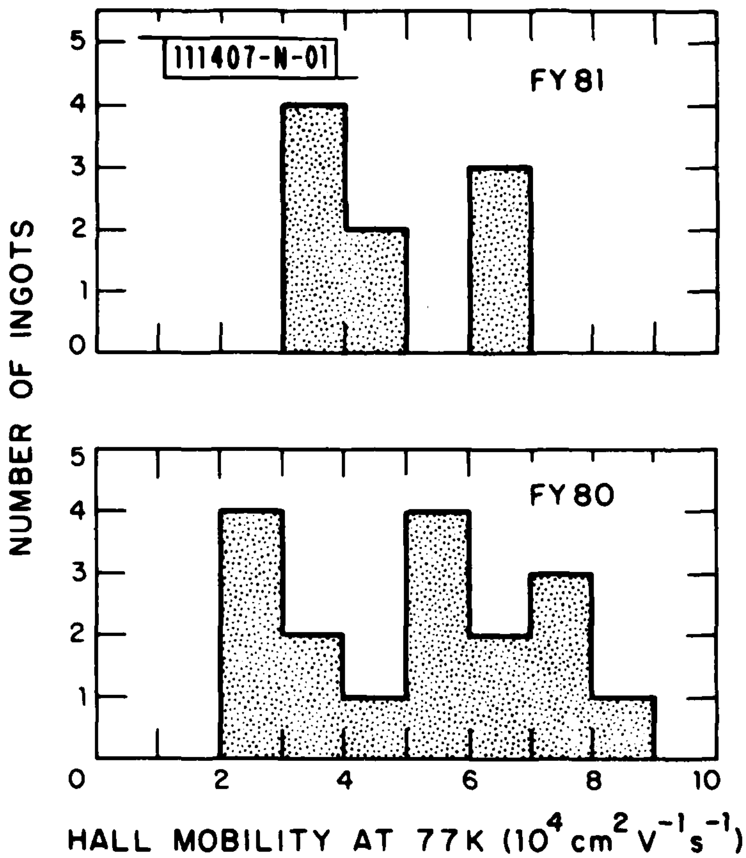

Fig. 2. Distribution of electron Hall mobilities at 77 K for InP ingots.

mobilities at 77 K (μ_{77}) for the nine measured ingots prepared during FY81 and also for the 17 measured ingots prepared during FY80. The values of μ_{77} range from 3.0×10^4 to 6.8×10^4 $cm^2V^{-1}s^{-1}$ and from 2.2×10^4 to 8.2×10^4 $cm^2V^{-1}s^{-1}$ for FY81 and FY80, respectively, compared with the highest reported value of 9.1×10^4 $cm^2V^{-1}s^{-1}$ for bulk InP.[1] For these ingots the carrier concentrations at 77 K (n_{77}, determined from the Hall coefficient by assuming a Hall factor of 1.0) range from 1 to 4×10^{15} cm^{-3} and from 5×10^{14} to 8×10^{15} cm^{-3} for FY81 and FY80, respectively, with μ_{77} generally decreasing as n_{77} increases.

For InP synthesis with In boat temperatures in the range from 1050 to 1180°C, Yamamoto, Shinoyama and Uemura[2] found μ_{77} to decrease with increasing boat temperature. As reported last year, however, we have found no correlation between μ_{77} and either the In boat temperature or the P reservoir temperature. Table I lists the P reservoir temperature (measured with a thermocouple tied very close to the one used for controlling the temperature at the end of the ampoule), the maximum temperature at the In boat, and the values of n_{77} and μ_{77} for the nine samples measured in FY81. The sample with the highest mobility (6.8×10^4 $cm^2V^{-1}s^{-1}$) was cut from an ingot synthesized with the highest boat temperature (1073°C).

In general the ratio of the electron concentration at 300 K (n_{300}) to n_{77} decreases with increasing μ_{77} as shown in Fig. 3. For the 19 samples shown on this figure with μ_{77} greater than 4×10^4 $cm^2V^{-1}s^{-1}$, only 3 exhibit n_{300}/n_{77} ratios greater than 1.1; for more than 30 samples with μ_{77} less than 4×10^4 $cm^2V^{-1}s^{-1}$, all but 8 exhibit n_{300}/n_{77} ratios greater then 1.1. The Hall factor dependence upon electron concentration and temperature probably accounts for the lower envelope of the data points, but compensation by acceptor impurities produces electron "freeze out" at 77 K and accounts for

TABLE I

SYNTHESIS TEMPERATURES AND ELECTRICAL PROPERTIES OF POLYCRYSTALLINE InP INGOTS

Ingot	Maximum indium boat temperature (K)	Phosphorus temperature (K)	n_{300} (10^{15} cm^{-3})	μ_{77} (10^4 cm^2 V^{-1}sec^{-1})
1*	1062	437	1.0	6.4
2	1057	438	4.2	3.3
3	1073	430	1.3	6.8
4	1045	460	3.3	3.8
5	1045	450	1.8	3.0
6	1043	468	1.8	4.7
7	1045	460	1.6	6.4
8	1040	458	2.3	3.9
9	1048	457	2.9	4.5

* Temperature profile for this ingot is shown in Fig. 1.

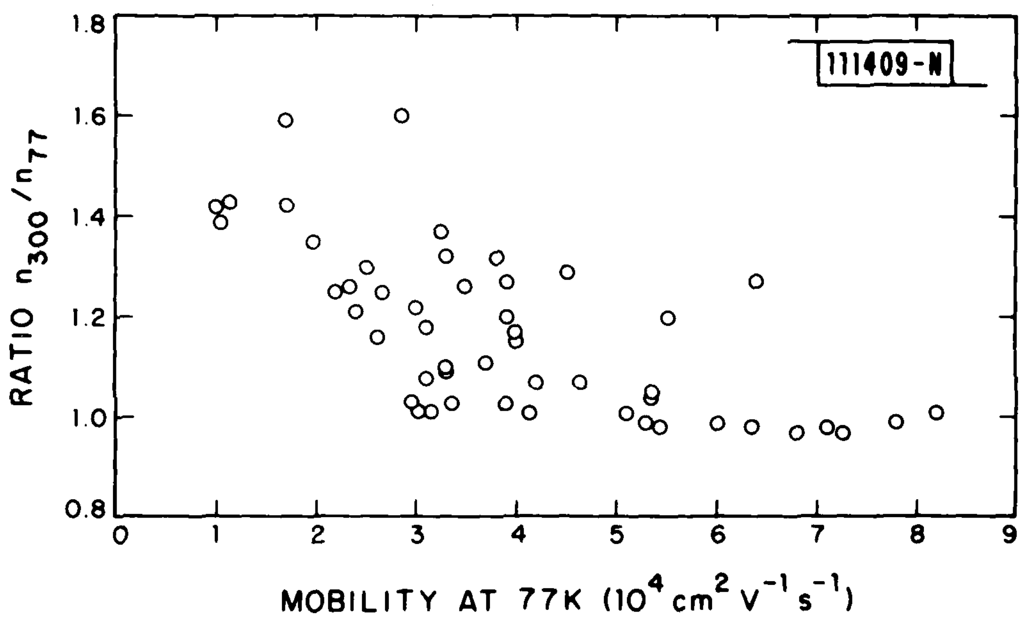

Fig. 3. Ratio of electron concentration at 300 K (n_{300}) to that at 77 K (n_{77}) vs Hall mobility at 77 K for InP ingots.

the data points lying above the lower envelope. Plotting the electrical data in this way may prove to be more helpful in determining which samples are compensated than plotting the mobility as a function of electron concentration in the conventional manner.

Most of the ingots with μ_{77} less than 3 x 10^4 $cm^2V^{-1}s^{-1}$ were synthesized prior to FY80, and spark source mass spectrographic analysis (SSMS) showed that increasing n_{77} and decreasing μ_{77} were correlated with increasing Si impurity concentration. The SSMS technique cannot be used to determine whether such a correlation exists for samples with μ_{77} greater than about 2.5 x 10^4 $cm^2V^{-1}s^{-1}$, since the Si concentration in these samples is too low to be determined accurately by this method. However, secondary ion mass spectrographic analysis (SIMS) of a sample (No. 1 of Table I) with μ_{77} = 6.4 x 10^4 $cm^2V^{-1}s^{-1}$ indicates that Si is still the major donor impurity. The SIMS data for this sample, which has n_{300} = 1.0 x 10^{15} cm^{-3}, are listed in Table II. The Si concentration is 1.3 x 10^{15} cm^{-3}, while S is present at a concentration of ~ 8 x 10^{14} cm^{-3}. The acceptor impurities are: Mg at 4 x 10^{14} cm^{-3}, Fe at ~ 7 x 10^{14} cm^{-3}, Cu and Cd each at ~ 4 x 10^{14} cm^{-3}. The SIMS studies also show that In inclusions and grain boundaries in the polycrystalline ingots contain concentrations of impurities several orders of magnitude higher than found in single-crystal areas. Auger electron spectrographic analysis shows that C appears above the detection limit (nominally 0.1 atomic percent) only at the In inclusions.

LEC GROWTH PROCEDURE

During FY80 our development of the LEC process for growing InP crystals was mainly devoted to reducing the probability of twinning, since twinning is almost always followed by polycrystalline growth. We found that a certain minimum temperature gradient at the liquid-solid interface was necessary to

TABLE II

SIMS ANALYSES OF INGOT AND LEC SAMPLES OF InP

Element	Ingot Sample No. 1 (cm^{-3})	LEC Sample (cm^{-3})
C	4.2×10^{15}	5.6×10^{15}
O	$< 3 \times 10^{15}$	4.2×10^{15}
Mg	4×10^{14}	$\sim 4 \times 10^{14}$
Si	1.3×10^{15}	2.3×10^{15}
S	$\sim 8 \times 10^{14}$	1×10^{15}
Cr	4×10^{14}	7×10^{14}
Mn	$< 4 \times 10^{14}$	4×10^{14}
Fe	$\sim 7 \times 10^{14}$	$< 7 \times 10^{14}$
Cu	$\sim 4 \times 10^{14}$	$< 4 \times 10^{14}$
Zn	$< 4 \times 10^{14}$	1×10^{15}
Cd	$\sim 4 \times 10^{14}$	$< 4 \times 10^{14}$
Sn	$< 4 \times 10^{14}$	$< 4 \times 10^{14}$

"<" = not detected

prevent twinning for any given set of the other growth parameters. During FY81, primarily by achieving a significant reduction in vibration at the interface, we have succeeded in growing twin-free crystals in vertical temperature gradients much smaller than those previously required in our system. Because of the decrease in radial temperature gradients that has accompanied the reduction in the vertical gradient, many of the crystals exhibit nearly zero dislocation density up to a diameter of ~ 20 mm, even without Zn or S doping.

Figure 4 is a scale diagram of the crystal growth assembly used in our high-pressure puller (A. D. Little Model HPCZ). To reduce vibration of the InP seed as it is raised and rotated, we have modified the water-cooled pulling rod and its rotation mechanism. A sturdier 1/2-in. diameter shaft driven by a direct coupling to a motor mounted on the ceiling has replaced the original 5/16-in. diameter pulling rod, which was rotated with a spur gear that meshed with and slid vertically along a spline gear. Vertical motion of the new rod is accomplished by a sliding spline in the drive shaft above the high-pressure seal. This seal, which originally consisted of a double O-ring, has been changed to a flat viton gasket. In addition, a teflon shaft wiper has been substituted for the original aluminum wiper, which abraded the pulling rod.

The InP seed is a (111)-oriented crystal that is mounted with the P face down on a Ni seed holder attached to the pulling rod. Seeds with square cross sections of 5 x 5 to 8 x 8 mm^2 or with a circular cross section of 15 mm diameter have been used. The diameter of the pyrolytic BN crucible has been increased to 6.8 cm and the crucible height slightly decreased to 3.8 cm. The weight of the polycrystalline InP charge has been varied between 175 and

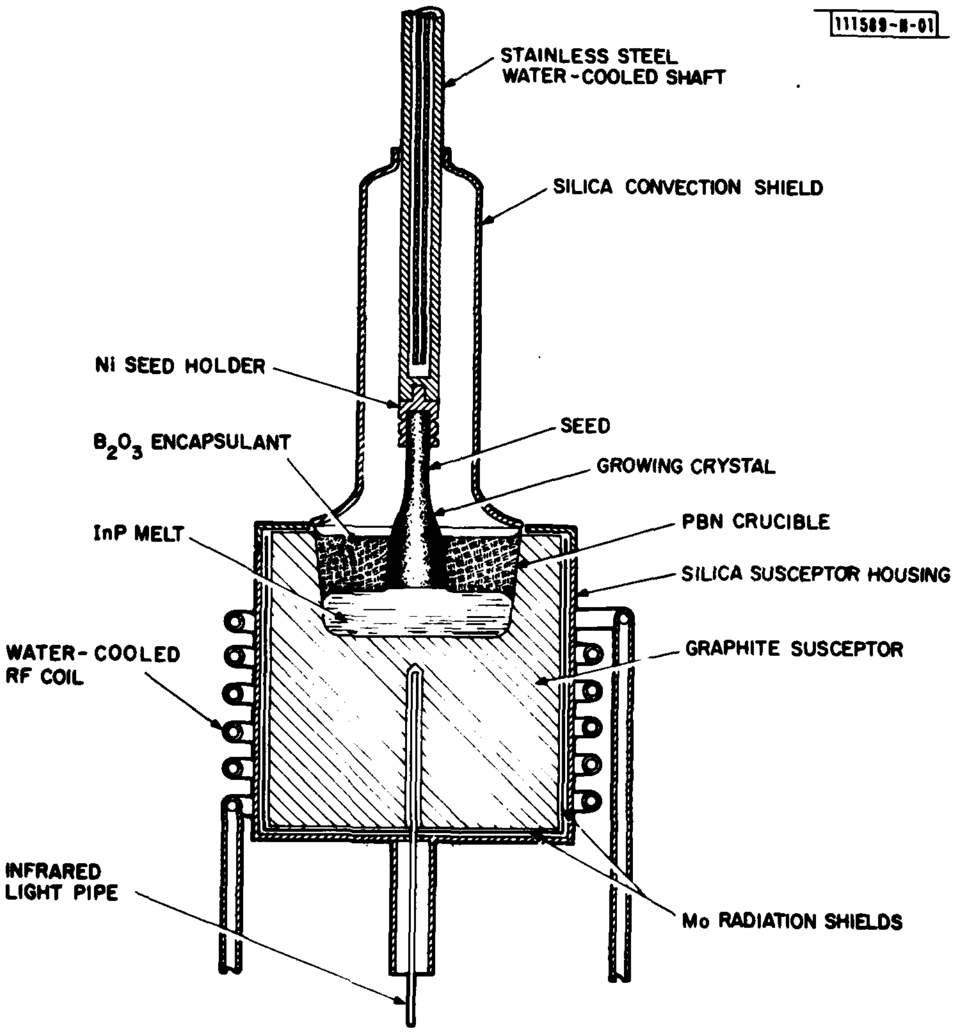

Fig. 4. Assembly for LEC growth of InP.

300 g, and that of the B_2O_3 encapsulant from 85 to 130 g (corresponding to an encapsulant layer thickness ranging from ~ 15 to 22 mm). The B_2O_3 has generally been pre-baked in vacuum at ~ 1200°C for ~ 8 h, but material purchased from RASA Industries and some from Johnson Matthey has been used as received.

The crucible is contained in a high-purity, high-density graphite susceptor, which is heated by means of an RF coil operated at 200 to 400 kHz. The RF current is monitored with a pickup coil and controlled by means of a three-mode solid state controller, and the relative temperature of the susceptor is monitored by using a photodiode detector to measure the infrared emission from the susceptor that is transmitted through a fused-silica light pipe. The Mo radiation shields shown in Fig. 4 are used to shape the temperature profile and to keep the RF coil and the walls of the pressure chamber from overheating, while the fused-silica convection shield resting on the crucible is used to reduce the convection currents in the high pressure gas surrounding the growing crystal. During FY80 the gas was a 10:1 mixture of high-purity Ar and He with a total pressure of 40 atm. Since installing the new pulling rod, the He has been eliminated and the Ar pressure decreased to values between 25 and 35 atm in order to reduce the temperature gradients. The susceptor (and therefore the crucible) and seed have been rotated in the same direction at rates varying from 2 to 30 rpm, and the pulling rate has ranged from ~ 15 to 30 mm/h.

LEC GROWTH RESULTS

In order to correlate InP crystal quality with the LEC growth parameters and to provide substrate material desired for a variety of applications, we have grown 96 boules during FY81. Of these, 26 were nominally undoped. The remainder were doped with Sn (29), Fe(19), S (11), Cd (8), Zn (2), or Cr (1).

Both electrical measurements and SIMS data show that the nominally undoped LEC crystals contain higher concentrations of impurities than the single-crystal regions of the ingots used as starting charges in the LEC puller. During the past two years electrical measurements have been made on 25 samples from nominally undoped LEC crystals and on 27 single-crystal samples from polycrystalline ingots. In all cases where measurements have been made both on a sample from a nominally undoped crystal and on a sample from the ingot used as charge material for that same crystal, the value of μ_{77} was lower for the LEC sample than for the ingot sample. Furthermore, values of μ_{77} exceeding 4×10^4 $cm^2V^{-1}sec^{-1}$ have been measured for 17 of the ingot samples but for only 3 of the LEC samples. Table II (Page 10) lists the SIMS results for the LEC sample with the highest value of μ_{77} that we have obtained so far. This sample contains significantly higher concentrations of C, O, Si, S, Cr, Mn and Zn than the ingot sample for which SIMS data are also given in Table II.

The fact that the LEC samples are higher in impurity content than the ingot samples is probably due at least in part to contamination that takes place in the process of converting ingot material to LEC single crystals. Another possible reason for the difference is that the impurities located in the grain boundaries and In inclusions of the ingots, which are not present in single-crystal samples cut from the ingots, will be released into the melt in the LEC puller. To the extent that these impurities are incorporated in the growing crystals, they will influence the properties of the samples from these crystals.

From the spatial distribution of dislocations in the InP crystals grown in FY80, as revealed by etch pit patterns on horizontal slices, it was clear that the dislocations in those crystals were produced by hoop stresses caused

by radial temperature gradients. During FY81 we have significantly reduced the dislocation density by decreasing the radial gradients while simultaneously establishing conditions necessary to maintain twin-free growth.

Since Shinoyama et al.[3] found that the dislocation density in LEC InP crystals was reduced by increasing the thickness of the B_2O_3 encapsulant layer, presumably because this decreased the radial temperature gradients, we increased the B_2O_3 thickness to ~ 2 cm. Temperature measurements made by Shinoyama et al.[3] on InP showed that increasing the B_2O_3 thickness also decreases the vertical temperature gradient; a similar relationship was inferred by von Neida, Oster, and Nielsen[4] from their experimental results on the LEC growth of GaP. To compensate for this effect, as a means of increasing the vertical gradient we increased the flow of heat transported upward through the seed by replacing the original BN seed holder with a Ni holder and by soldering the seed directly to the new holder with In. Nevertheless, with the increased B_2O_3 thickness the yield of untwinned crystals was nearly zero until we installed the new pulling rod to reduce vibration. Since that change the yield of twin-free crystals has been quite high even though the vertical gradient has been further reduced by eliminating the He gas, reducing the Ar pressure to 30 or even 25 atm, and raising the RF coil with respect to the susceptor.

Our efforts to measure the temperature gradients in the LEC puller by using thermocouples have been unsuccessful due to RF induced noise. However, we estimate that the vertical temperature gradient along the growing crystal within the B_2O_3 layer is now less than half the gradient that was established under the standardized conditions that we used in FY80 for twin-free growth. This estimate is based on the observation that the runoff of B_2O_3 from the growing crystal is greater now than it was under the former conditions. This

indicates that the surface temperature of the crystal at the B_2O_3-gas interface is higher now than before.[3] Therefore the temperature difference between this interface and the melt surface is less than before, although the thickness of the B_2O_3 is about twice as great.

Etch pit studies show that the first-to-freeze portions of twin-free crystals grown under the new conditions generally have very low dislocation densities (less than a few times 10^3 cm^{-2} and sometimes nearly zero) until their diameter reaches a threshold value of about 20 mm, even when they are nominally undoped or doped with impurities other than Zn or S. When the diameter increases beyond this threshold, the critical resolved shear stress is exceeded by the hoop stresses, causing a rapid increase in dislocation density. For the larger diameters, the dislocation densities are of the order of 10^4 cm^{-2}.

Necking the crystal down just below the seed to reduce the dislocation density, as suggested by Shinoyama, et al.,[3] has proved unnecessary if the radial gradient is low enough. For example, one growth run with a cylindrical seed 15 mm in diameter yielded a 3 cm length of nearly dislocation-free, nominally undoped crystal of the same diameter without any necking in at all.

To supplement the etch pit studies, x-ray topography studies have been made on longitudinal sections (generally with (110) surfaces) cut from doped LEC crystals and polished to a thickness of 150 to 300 μm. Transmission projection topographs confirm that the material is nearly dislocation-free up to a diameter of about 20 mm. The most striking feature of the topographs is the large number of prominent growth striations that they reveal. Such striations are shown clearly in Fig. 5, a topograph taken on a S-doped (110) sample with Ag radiation and (111) diffraction vectors. The discontinuities observed in many of the striations are presumably due to

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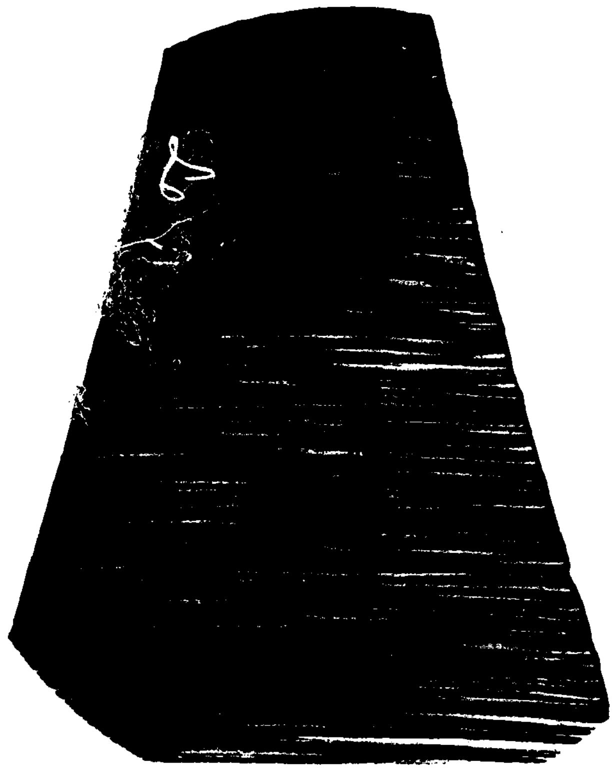

Fig. 5. X-ray topograph of longitudinal (110) slice from a S-doped LEC InP crystal.

remelting, which may be caused by asymmetric radial temperature gradients in the melt. These gradients may come about because the termination of the RF coil at top and bottom destroys the radial thermal symmetry, although the susceptor rotation should reduce this effect.

We have made free-carrier absorption measurements on LEC crystals doped with S, Sn or Cd to concentrations of the order of 10^{18} cm^{-3} which show that the growth striations are associated with large variations in dopant concentration. These measurements are performed by determining the variation in infrared transmission as a longitudinal section of a crystal is scanned across the focused beam of a CO_2 laser. The sample thickness is chosen to give transmission values small enough so that interference effects due to multiple reflections of the laser beam are negligible. Figure 6 is a recorder trace, obtained by a vertical scan of an x-ray topography sample 160 μm thick, showing the variation in transmission with distance along part of a S-doped crystal, which is n-type. An electron concentration scale, shown as the right-hand ordinate of Fig. 6, was constructed by using the data of Walukiewicz, et al.[5] for the absorption of CO_2 laser radiation by InP as a function of electron concentration. The apparent concentration in the sample varies from about 2.4×10^{18} cm^{-3} to more than 3.5×10^{18} cm^{-3}. The actual variation may be even greater, since the laser spot size (~ 30 μm) may have been too large for completely resolving the transmission peaks and valleys. Similar relative variations in carrier concentration are revealed by the transmission measurements on Sn-doped crystals, which are also n-type, and on Cd-doped crystals, which are p-type. Since no data have been published on the free-carrier absorption of p-type InP, in order to evaluate the carrier concentrations in the Cd-doped crystals we have initiated a study to determine the absorption coefficient at 10.6 μm as a function of hole concentration.

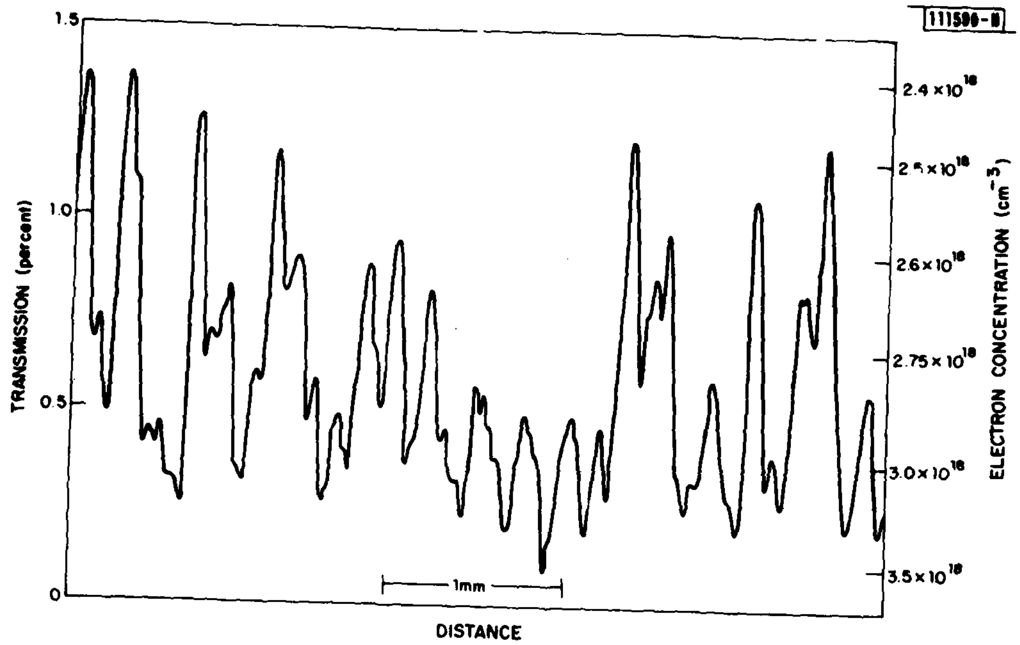

Fig. 6. Recorder trace showing transmission of CO_2 laser radiation vs distance along vertical direction for S-doped LEC InP crystal.

The data obtained so far are plotted in Fig. 7. These data show that holes have a much larger cross section than electrons. For example, for a carrier concentration of 1 x 10^{18} cm^{-3} the absorption coefficient at 10.6 μm is about 500 cm^{-1} for p-type material, compared with about 75 cm^{-1} for n-type material.

The sharp, irregular variations in dopant concentration shown by the transmission measurements are probably produced by random convection currents in the melt, which could also contribute to remelting. As a result of such currents, the liquid forming the boundary layer at the growth interface is replaced by liquid from the bulk of the melt. Since in the absence of convection the impurity concentration in the boundary layer liquid is increased by rejection of impurity atoms from the growing crystal, replacement of this liquid would cause a temporary decrease in the impurity concentration in the melt at the interface and a corresponding decrease in the concentration incorporated into the crystal. Longitudinal variations in dopant concentration can also be produced by the intermittent formation of horizontal growth facets, which would cause the impurity atoms within the boundary layer to be trapped in the crystal by rapid lateral growth. However, it is unlikely that this mechanism contributes significantly to the present striations, since in the x-ray topographs these striations do not contain straight-line segments, which would occur if the interface had the planar shape characteristic of facet formation.

To obtain additional evidence concerning the possible role of facet formation, we have used $(\bar{1}00)$ seeds for the growth of 4 Sn-doped LEC boules, since for other III-V compounds facet formation is much less probable for (100) growth than for (111) growth. Only one of the (100) boules was untwinned, suggesting that the probability of twinning is higher for this growth orientation. For the untwinned crystal the relative longitudinal

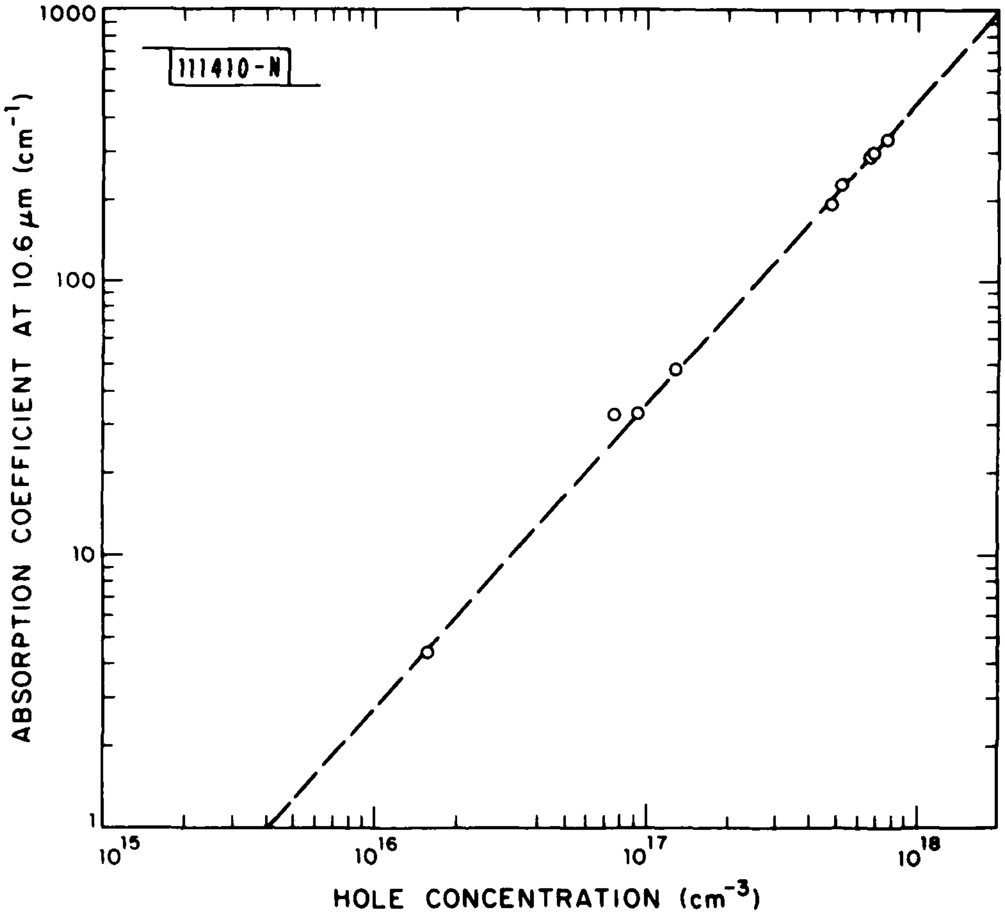

Fig. 7. Absorption coefficient at 10.6 μm vs free hole concentration for p-type InP samples.

variation in dopant concentration is comparable to that observed for the (111) crystals, again indicating that facet formation does not contribute significantly to the striations.

FUTURE WORK

During FY82 our efforts to improve the quality of LEC InP crystals will be directed toward reducing three types of imperfections -- residual impurities, dislocations, and dopant striations -- without increasing the occurrence of twinning.

The SIMS data show that impurities are concentrated in the grain boundaries and In inclusions of the InP ingots used as charge material for LEC growth. With the objective of reducing the residual impurity content of the LEC crystals, we shall therefore attempt to increase the grain size and reduce the amount of second-phase In in the ingots by modifying the synthesis conditions -- in particular, by increasing the temperature gradient at the liquid-solid interface and by reducing the rate of solidification. We shall also attempt to decrease the impurity content of the ingots by vacuum baking the In at high temperature prior to InP synthesis. Kubota and Sugii[6] have reported that pre-baking In at 900°C results in an increase in μ_{77} due to the removal of O, S, and perhaps Si.

The dislocations in LEC InP crystals are generated primarily by hoop stresses resulting from the radial temperature gradients that are present just above the seed-melt interface. With the objective of increasing the maximum diameter to which crystals can be grown dislocation-free, we shall therefore attempt to modify the growth conditions in a manner that will further reduce these radial gradients without increasing the probability of twinning. Since we were able to accomplish this during the past year by increasing the thickness of the B_2O_3 layer while reducing the vibration in the puller to

permit twin-free growth in lower vertical gradients, we plan to extend this approach by improving the design of the susceptor support shaft to further reduce vibration.

Since the growth striations in the LEC crystals are probably due to random convection currents in the melt, in order to decrease the inhomogeneities in dopant concentration we will attempt to reduce the convection currents. As one approach we are investigating the utilization of fused-silica and pyrolytic BN baffles in the growth crucible. A second approach is suggested by results on the Czochralski growth of Ge crystals reported by Martin, Witt, and Carruthers,[7] who found that the convection currents and therefore striations are dramatically reduced by using a heat pipe to establish more nearly isothermal conditions in the melt. Since the heat pipes now available cannot withstand the high pressures employed in the LEC puller, as an alternative method for decreasing temperature gradients in the melt we will investigate the replacement of the present graphite susceptor by a liquid Ga susceptor, with the growth crucible immersed directly in the liquid metal.

ACKNOWLEDGEMENTS

The author is grateful to J. V. Pantano, E. J. Delaney and M. S. Taylor for their expert technical assistance, to E. B. Owens for the spark source mass spectrographic analyses, to M. C. Finn for the Auger analyses and to R. J. Markunis (Department of Electrical Engineering and Computer Science, M.I.T.) for the SIMS analyses.

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER ESD-TR-81-301	2. GOVT ACCESSION NO. AD-A110508	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) InP Materials		5. TYPE OF REPORT & PERIOD COVERED Annual Technical Summary Report 1 October 1980 – 30 September 1981
		6. PERFORMING ORG. REPORT NUMBER None
7. AUTHOR(s) Gerald W. Iseler		8. CONTRACT OR GRANT NUMBER(s) F19628-80-C-0002
9. PERFORMING ORGANIZATION NAME AND ADDRESS Lincoln Laboratory, M.I.T. P.O. Box 73 Lexington, MA 02173		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Program Element No. 61102F Project No. 2306
11. CONTROLLING OFFICE NAME AND ADDRESS Rome Air Development Center Griffiss AFB, NY 13440		12. REPORT DATE 30 September 1981
		13. NUMBER OF PAGES 30
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) Electronic Systems Division Hanscom AFB Bedford, MA 01731		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

None

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

InP crystal growth   InP Synthesis

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report covers the work on InP materials carried out with support of the Department of the Air Force during the period 1 October 1980 through 30 September 1981. A part of this support was provided by the Rome Air Development Center. LEC crystals that are nearly dislocation-free up to a diameter of ~20 mm have been grown without Zn or S doping. X-ray topography and CO_2 laser transmission studies have shown that doped LEC crystals contain prominent growth striations that are associated with abrupt changes in dopant concentration.

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